Optical scattering and microstructure of gold and platinum coatings

G.A. Al-Jumaily, J. M. Bennett*, D. R. Coulter and N. A. Raouf
Jet Propulsion Laboratory
California Institute of Technology
4800 Oak Grove Drive
Pasadena, CA 91109

ABSTRACT

The microstructure of gold and platinum coatings deposited using thermal evaporation and ion beam deposition processes onto super polished fused silica substrates have been examined. Coatings deposited using ion beam deposition have lower optical scattering and related surface microroughness.

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* Michelson Laboratory, Naval Air Warfare Center, China Lake CA.

I. INTRODUCTION

Metallic coatings are widely used in optical applications such as astronomical telescopes where broadband reflectivity is necessary. Gold (Au), silver (Ag) and aluminum (Al) are the most commonly used coating materials, Ag has the highest reflectance at visible and IR wavelengths and shows good adhesion to optical substrates. However, Ag coatings readily reacts with sulfer forming silver sulfide which is commonly called tarnish thus limiting their usefulness. Al has a relatively high reflectance throughout the visible and IR region of the spectrum and also shows good adhesion properties. Al coatings are considerably more stable than Ag coatings. However, a native oxide is also formed on this material upon exposure to the atmosphere which limits its usefulness in the vacuum ultraviolet. Au has excellent reflectance in the IR and is very environmentally stable. Au coatings, however, have low reflectance in the visible region of the spectrum and, in general, exhibit poor adhesion which makes it necessary to utilize a thin layer of chromium or other metal to improve bonding.

Platinum (Pt) is a material that has not traditionally been used in reflecting coating applications. This is due in part to its high melting point which makes it difficult to apply by traditional evaporation techniques. It can, however, be sputtered quite easily. Pt has good reflectivity in the visible and IR region of the spectrum, does not form an oxide on exposure to the atmosphere and is anticipated to show good adhesion to optical substrates. Sputtered Pt coatings may represent a good alternative to the traditional metallic coating materials resulting in improved reflectivity, stability and acceptable reflectance.

In addition to providing good reflectivity and adhesion, reduction of optical scattering from mirror surfaces is another major concern for space-based telescopes in particular. On the ground, without adaptive optics, the atmosphere is the dominate source of scatter. In space or in ground-based adaptive optics systems, scattering from the optics themselves limits the telescope's ability to resolve faint objects close to much larger and brighter sources. Scattering from metal-coated mirrors is caused by the surface roughness and the optical figure of the substrate and the microstructure of the metal coatings. Surface roughness of the substrate can be minimized by using a single crystal substrate such as silicon or superpolished glassy materials such as fused silica. Figure errors can be reduced to very low levels by means of standard polishing or ion beam figuring. Consequently, scattering from the coatings themselves becomes the dominant contributor to the overall scatter.

Coating microroughness is related to the columnar growth processes in thin films. In previous work, ion beam deposition techniques have been used to modify the columnar growth process and reduce the microroughness of thin films.[1 -6] [n this paper, results are presented on the surface roughness and scattering of gold and platinum coatings deposited by four different techniques onto superpolished fused silica substrates. The coating processes employed were thermal evaporation, ion assisted deposition, ion beam sputtering, and ion assisted sputtering.

II. SAMPLE PREPARATION

Superpolished fused silica flats were purchased from General Optics in Moorpark, CA. The flats were 1" in diameter and showed surface roughness values of < 1Å as measured by optical interferometry and profilometry.

Gold and platinum coatings approximately 100 nm thick were deposited in a 60 cm box coater at Barr Associates in Westford, MA. The coating system was cryogenically pumped to a working pressure of $2x10^{-6}$ Torr. The substrates were not heated and bias voltage was not applied to the substrate holder. The deposition system was equipped with a 2.5 cm Kaufman ion source, a 7.5 cm dc sputtering source and a resistance heated evaporation source. Ultra high purity argon was used in the ion source at a pressure of 2x10-4 Torr. For sputtering, the total pressure was 1X10-3 Torr. Deposition rates and film thickness were monitored using a crystal rate monitor as well as an optical monitor.

As indicated previously, films were deposited by evaporation, ion assisted deposition (IAD), sputtering and ion assisted sputtering (IAS). In each case, the substrates were ion-cleaned prior to coating with 10 mA of 300 eV argon ions for about three minutes. Evaporated Au coatings were deposited with and without an underlayer of chromium. In the IAD case, the evaporated Au coatings were bombarded with 5 mA of 300 eV argon ions from the Kaufman ion source during deposition. In the IAS case, the sputtered Au coating was bombarded using the same beam parameters described above.

Pt coatings were deposited using sputtering and IAS only. Deposition conditions for the Pt coatings were similar to those of the sputtered and IAS Au coatings. No attempt was made to deposit Pt coatings using evaporation or IAD because of the high melting temperature of the metal.

III. CHARACTERIZATION TECHNIQUES

Au and Pt coated superpolished flats have been characterized using a variety of diagnostic techniques and instrumentation. The characterization included the following measurements:

1. Spectral Reflectance

   The spectral reflectance of the coated substrates was measured using a dual beam spectrophotometer with a reflectance attachment. Since both Au and Pt reflect well in the IR, the measurement range of interest was near the absorption edge for Au, from 500 to 800 nm.

2. Angle-Resolved Optical Scattering

   The coated superpolished fused silica substrates were examined using an angle-resolved optical scatterometer (AROS) at Sandia Systems. The AROS system uses a He-Ne laser (λ=632.8 nm) as a source. The laser beam which is focused onto the detector has an area of about 1 mm onto the sample. Hence, measurement is done in the far field. The scattered light intensity is measured in the plane of incidence using a photomultiplier tube. The spatial wavelength range of the system determined by the system geometry and the source wavelength is 0.7 to 72 µm. Detailed description of the system can be found elsewhere. [2-4]

   The scattered light characteristics of the surface can presented as the bi-directional reflectance distribution function (BRDF) or the power spectral density (PSD). A value for the RMS roughness is obtained by integrating the PSD curve. [7]

3. Stylus Profilometry

The stylus profilometer measures the roughness of surfaces by moving a diamond tip across the surface. The movement of the tip is transformed into an electrical signal, The spatial wavelength range is determined by the tip diameter, the scan length and the sampling frequency. Coated substrates have been characterized using a stylus profilometer which has a 1 mg loading, 1.0 pm tip radius, and 100 and 1000 μm scan lengths. Detailed description of the technique and its application to surface roughness measurement can be found in Ref. 8.

4. Scanning Tunneling and Atomic Force Microscopy

A scanning tunneling microscope (STM) and an atomic force microscope (AFM) have been used to examine the surface microroughness of uncoated and coated substrates. The STM technique profiles surfaces using a tungsten stylus which senses the electron tunneling current between the surface and the tip. Consequently, the surface under examination must be conductive. The STM technique is used to characterize coated substrates. The AFM technique is similar to the STM except that the microscope has a silicon nitride tip on the end of a cantilever arm which is deflected by atomic forces between the surface and the tip. Since the AFM can be utilized on both conducting and non-conducting surfaces, it has been used to characterize both coated and uncoated substrates. Three dimensional STM and AFM data are collected and digitized as the as the stylus moved across the surface, The RMS surface roughness is determined by calculating the standard deviation of the out-of-plane data around the most probable plane of the surface as determined by the measurement.

5. Optical Profilometry

Coated and uncoated samples were characterized using a Chapman MP2000 optical profilometer. The instrument uses two orthogonally polarized beams to profile the surface. Detailed description of the instrument can be found in Ref. 9.

IV. EXPERIMENTAL RESULTS

a. Au Coatings

The spectral reflectance of Au coatings deposited by the four different techniques described previously has been measured over the spectral range from 500 to 800 nm. Results indicated that in general, sputtered coatings had a higher reflectance than the evaporated films either with or without ion bombardment Results also showed that ion bombardment of the evaporated coatings caused a slight increase in reflectance while similar treatment of the sputtered coatings caused a slight decrease. For example, the reflectance at 600 nm for the evaporated, IAD, sputtered and IAS coatings were 85, 87, 93 and 90, percent, respectively.

Plots of the PSD characterized using the AROS system for four Au coated superpolished fused silica substrates are shown in Fig. 1. It should be noted that the IAD sample, in addition to having a higher reflectivity has lower optical scattering than the evaporated sample. The sputtered Au sample, however, has the lowest optical scattering as well as the highest reflectivity observed in this study. Ion bombarding the sputtered Au coating (IAS) has introduced a shoulder in the PSD plot, indicating an increase in scattering at angles near the specular beam.

Values of the RMS roughness for the four Au coated fused silica substrates have been calculated from the optical profilometry data using three spatial filters of 0.08, 0.8, and 2.5 mm, These results are given in Table 1.

Table 1

RMS roughness for gold coated fused silica flats calculated from optical profilometry data with spatial filters of 0.08, 0.8 and 2.5 mm

Sample	RMS Roughness (Å)		
	0.08	0.8	2.5
Evaporated Au	7.0	24.0	48.2
IAD Au	4.4	15.3	27.9
Sputtered Au	0.2	0.5	3.2
IAS Au	0.2	1.1	9.9

It should be noted that the calculated rms roughness for all surfaces depends strongly on the spatial bandwidth of the measurement. An improvement (reduction) in the rms roughness is observed for all spatial bandwidths in the IAD coatings compared to evaporated coatings. Sputtering, however, yields much smoother coatings than evaporation, Also, note that the difference in the rms roughness between the sputtered and the IAS coatings depends on the bandwidth of the calculation. For example, the rms roughness for both samples is the same using the 0.08mm filter, while the roughness of the IAS coating is twice that of the sputtered coating using the 0.8mm filter and three times larger using the 2.5mm filter. The increase in the rms roughness using progressively coarser filters indicates that all of the films have significant low frequency features. In the case of the IAS coating, ion bombardment results in an order of magnitude increase in roughness between the calculation using the 0.8mm filter and the one using the 2.5mm filter. This suggests that the ion bombardment induces long range features which is consistent with the observation of a shoulder in the optical scattering data at low spatial frequencies.

The uncpated fused silica substrates were characterized using the optical profilometer. The RMS roughness of ther uncoated was much higher than the sample coated with sputtered Au. The apparent high roughness of the uncoated substrate is caused by bulk and reflection from the back surface.

The scattering and profilometry data provided indirect measures of the surface roughness of the gold coatings applied by the four techniques.

Assuming that the chemical nature of the surfaces of all the coatings are identical, then the STM technique should allow for direct observation and measurement of the roughness. Figure 2 shows four STM micrographs of Au coated fused silica substrates. The relevant spatial bandwidth for these measurements was 2.5nm to 1pm. The corresponding RMS roughness values of the samples are listed in Table 2.

Table 2
RMS roughness of Au coated superpolished fused silica substrates obtained using the STM technique

Surface	RMS (Å)
Evaporated	42
I AD	49
Sputtered	26
IAS	23

The micrographs in Figure 2 and the calculated RMS roughness values given in Table 2 are consistent with previously discussed data which indicate that sputtered films are in general, smoother than evaporated films. The RMS roughness values as measured by STM are larger than those obtained using other techniques, presumably because of the inclusion of much higher spatial frequencies in the measurement. The STM data show little difference between the sputtered and IAS coatings. The shoulder observed in the scattering data from the IAS coating can be associated with features whose spatial period is >1μm and thus would not be seen in the STM data.

b. Au/Cr

To examine the influence of a thin Cr underlayer on the microroughness of Au coatings, four samples were prepared. Two Au coatings with and without a Cr underlayer were deposited using sputtering. Two similar samples were deposited using the IAS technique. Samples of the bare fused silica (FS) substrates and the coated substrates were examined using the stylus surface profilometer and the AFM. The scanned area for the AFM is

1pm x 1pm and the scan length for the stylus is 100 μm. Values of the rms roughness obtained using both techniques are listed in Table 3.

Table 3

Values of the RMS roughness for Au coatings showing the effects of a Cr underlayer. The scanned area for the AFM is 1μm x 1μm and the scan length for the stylus is 100 μm.

Surface	RMS Roughness Å)	
	Stylus	AFM
Uncoated FS	0.72	1.6
Sputtered Au	3.33	16.1
Sputtered Cr/Au	3.11	12.3
IAS Au	3.11	14.6
IAS Au/Cr	2.75	10.4

Results indicate that the presence of a Cr underplayer causes a small reduction in the microroughness of both sputtered and IAS Au coatings. It is also apparent that the roughness of the sputtered Au and IAS Au coatings measured by the AFM technique is less than that measured by the STM technique (see Table 2) although the relative values are similar. This is likely due to the fact that the variation in electron tunneling current and interatomic forces between the surface and the stylus tip vary differently with distance. In addition, the probe tungten probe used in the STM technique is alomst atmically sharp whereas the silicon nitride tip used in the AFM has a larger radius. The roughnnss values measured using a larger tip are smaller than those obtained using a sharper tip. Figure 3 shows two AFM micrographs of IAS Au with and without Cr underlayer. Two AFM micrograms are shown in Fig. 4 to illustrate the effects of ion bombardment on the surface microstructure of Au/Cr coatings. It should be noted that ion bombardment causes the grains to be smaller.

c. **Pt Coatings**

The microroughness of Pt coatings deposited using sputtering and IAS techniques onto superpolished fused silica substrates have also been examined. The rms roughness values for Pt coatings obtained from the stylus and AFM scans as well as the AROS system are presented in Table 4. The scanned area for the AFM is 1pm x 1pm, the laser beam spot size on the sample is 3 mm and the scan length for the stylus is 100 pm.

Table 4
Values of the RMS roughness for Pt coatings obtained using the stylus profilometer, AFM, and angle resolved optical scatterometer

Surface	RMS Roughness (Å)		
	Stylus	AFM	AROS
Sputtered Pt	1.52	10.2	6
IAS Pt	1.28	8.0	7

Comparing the results in Table 4 to those in Table 3 clearly indicates that the Pt coatings are smoother than Au or Au/Cr coatings applied by similar techniques. As in the case of the Au coatings, the AFM and the stylus results indicate that the IAS Pt coatings are smoother than the sputtered coatings. The measurement by the AROS system indicates a slight increase in roughness of Pt coatings deposited by the IAS technique. There is, however, no indication of structure in the PSD curve for the IAS Pt coatings as was observed in the case of the IAS Au coatings. Consequently, we believe that the sputtered and IAS Pt coatings are essentially identical over the lower spatial frequencies. Figure 5 shows two AFM micrographs of the sputtered and IAS Pt coatings on fused silica.

The effects of substrate material on the surface microroughness of sputtered Pt coatings were examined, Two sputtered Pt coatings were deposited onto supepolished fused silica and Zerodur substrates. The STM micrographs of the two samples are sheen in Fig 6. The RMS roughness of the Pt coating deposited onto Zerodur calculated from the stylus profilometer and STM measurements were 1.32 and 24.1 Å, respectively. However the RMS roughness of Pt coatings on fused silica obtained using the STM technique was 17.2 Å.

V. DISCUSSION

The surface roughness and microstructure of gold and platinum coatings deposited onto superpolished fused silica substrates using various deposition processes have been examined. Results presented above indicate that ion beam deposition techniques (IAD, sputtering and IAS) introduce significant changes in metallic coating microstructure. In general, ion bombardment resulted in significant reduction in the surface microroughness of coatings as compared to thermal evaporation. Sputtered Au films showed lower surface microroughness than evaporated and IAD coatings. However, increased ion bombardment (IAS) caused an increase in the surface roughness (at lower spatial frequencies) as measured with the AROS system. Platinum coatings deposited using sputtering and ion assisted sputtering have lower surface roughness as compared with gold films. The IAS Pt coatings show essentially no change in rms roughness as compared to sputtered coatings at low spatial frequencies as a result of ion bombardment when measured with the AROS system and a slight decrease at high spatial frequencies when measured by the stylus profilometer or AFM.

The difference in the way this process affects the microstructure of Au and Pt may be explained by referring to the zone models of Movchan and Demchishin [9] and Thornton [10]. These models relate the microstructure of thin film coatings to the temperature of the substrate and the melting point of the coating material. The temperature of the substrate is usually used as a measure of the kinetic energy of the adatoms on the surface and can be varied by simply heating the coated surface. In the case of the IAS technique, the energy of the atoms on the surface is also related to the ion bombardment current and energy. Increasing the kinetic energy of the adatoms leads to increased surface mobility and re-evaporation. High surface mobility allows the adatoms to seek low potential energy sites, and the loosely bonded adatoms to re-evaporate. Hence, increasing kinetic energy increases the packing density of the coating which leads to decreasing microroughness. Further increasing the surface temperature or kinetic energy relative to the coating melting point leads to re-crystallization which frequently results in increased optical scattering. The melting temperature of Pt is 1772°C compared to 1064°C for Au. Thus, it may well be that the additional energy associated with the ion bombardment in the IAS technique results in some crystallization of the Au films which results in increased scattering. The crystallization of metal coatings under ion bombardment has been reported by Howngbo et al.[11]

IV. CONCLUSIONS

Optical scattering and microstructure of Au and Pt coatings deposited using thermal evaporation and ion beam processes have been examined. Several characterization techniques were employed to examine the effects of deposition **process** parameters on the microroughness of coatings. Results indicate that ion beam deposited coatings are smoother than evaporated coatings and Pt coatings are smoother than Au coatings deposited under the same conditions.

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Figure Captions

1. PSD of four gold coatings deposited onto superploshed fused silica substrates. Plot 1 represents evaporated Au, 2 represents IAD Au, 3 represents sputtered Au, and 4 represents IAS Au.

2. Four STM micrograms representing the surface microroughness of Au coatings deposited using evaporation, IAD, sputtering and sputtering with ion bombardment (IAS).

3. Two AFM micrographs showing the effects of a Cr underlayer on the surface microstructure of IAS sputtered Au.

4. Two AFM micrographs showing the effects of ion bombardment on the surface microstructure of sputtered Au/Cr.

5. Two AFM micrographs showing the effects of ion bombardment on the surface microstructure of sputtered Pt coatings,

6. Two STM micrographs showing the surface microstructure of sputtered Pt coatings deposited onto superpolished substrates os fused silica and Zerodur.